DISCLOSURE LETTER OUTLINE

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Building: K-1 Room: 4A16
Date: January 1999

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SUBJECT: PATENT DISCLOSURE LETTER

on: Method for Fast GPC Separation with Molecular Weight Determination for Combinatorial Chemistry.

1. OBJECT OF INVENTION (e.g., problem, opportunity, prior art)

Traditionally, measurement of molecular weight of synthetic polymers has required times of up to 40 minutes for separation and molecular weight (MW) determination using banks of columns with a range of packing porosities. The parallel synthesis of polymers using the multiplicity of catalysts that can be prepared by combinatorial synthesis methods can generate samples at a rate sufficient to exceed the capacity of reasonable multiples of traditional instruments.

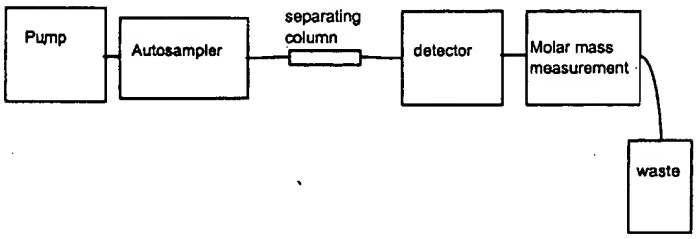
This invention describes a process to separate polymers from small molecules and subsequently determine the absolute MW averages using a combination of detectors. The invention also describes a device to measure molecular weight by light scattering simultaneously with the determination of concentration. The method of the invention and the device are intended to be used for rapid catalyst discovery and reaction condition optimization.

2. DESCRIPTION OF INVENTION

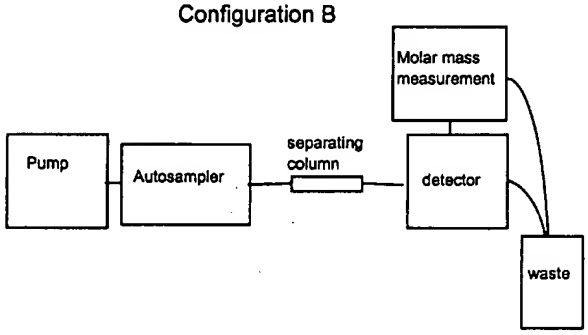
The method of the invention is to assemble a liquid chromatograph utilizing a short column or columns packed with a robust size exclusion material that separates small monomer and reagent molecules from oligomers and polymer. The concentrations of the components can be detected using existing liquid chromatography detectors such as UV absorbance, differential refractive index, ultrasonic, infrared absorbance, fluorescence and others known to those skilled in the art. The weight average molecular weight of the separated higher molecular weight fraction (HMWF) consisting of polymers and oligomers is then determined by employing a light scattering detector either in series with the concentration detector or by diverting the HMWF to an off-line cell, that is the subject of the device in this invention, for simultaneous concentration and light scattering determinations Block diagrams of two embodiments of the method of the invention are shown in Figure 1.

Block Diagrams of Fast GPC Instrumentation Options

Configuration A



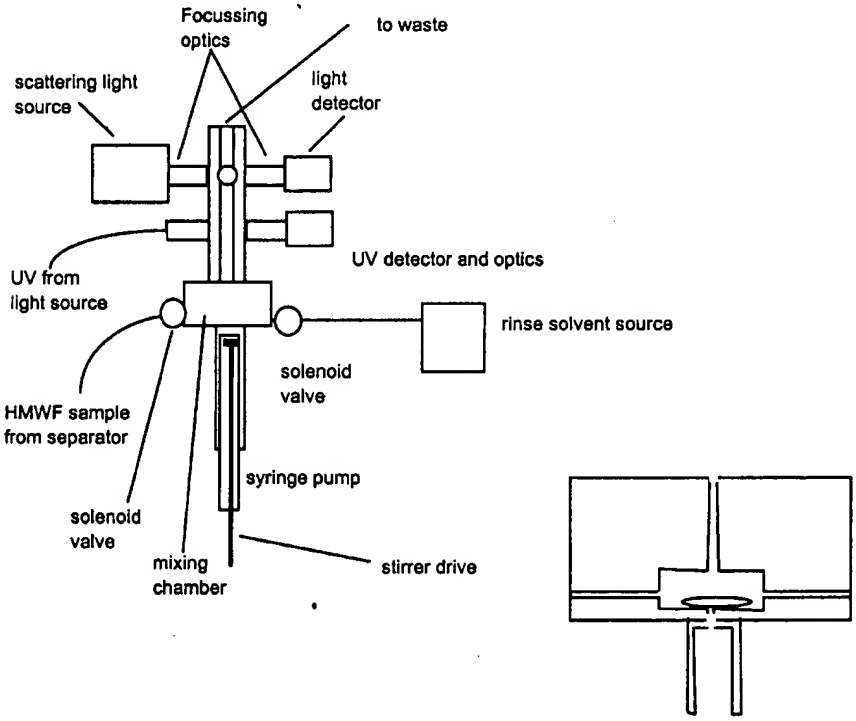
Configuration B

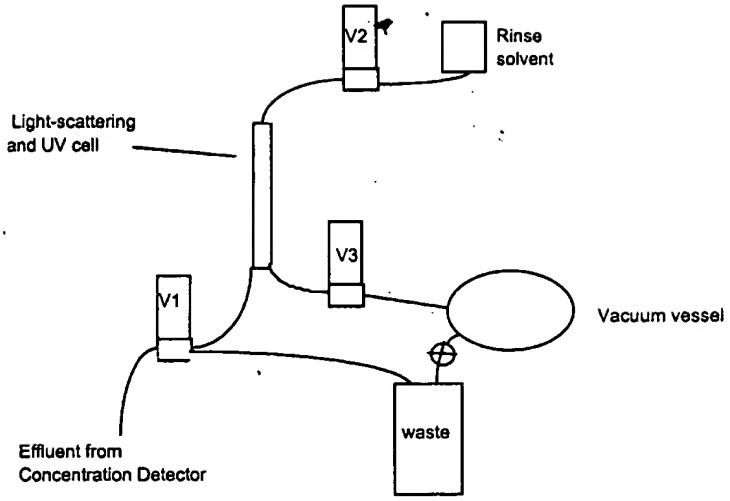


The in-line light scattering instrument can be obtained commercially from at least two sources (Wyatt Technologies and Viscotek Corp.)

The off-line light scattering cell is a square core quartz cell with a cell volume capable of containing the entire HMWF peak. In the case of the example below, the volume requirement is about 300 microliters. The cell includes a means of introducing the isolated HMWF, a means to stir the cell contents to insure concentration homogeneity, a means of removing the sample to waste after measurement and a means to rinse the cell. On one face of the cell, a visible or IR light beam from a laser will be passed through the cell volume. In another portion of the same cell face a collimated light beam from narrow wavelength UV source will be passed through the cell. The UV light may be

conveniently brought to the cell using fiber optic transmission lines. The non-scattered light from the laser beam that passes through the cell shall be collected by appropriate optics and the intensity determined by a photo-detector. Light that is scattered at close to 90° from the entry beam shall be collected by one or two sets of the same optics and photo-detector used for the non-scattered light. The ratio of the scattered and non-scattered light shall be used along with externally generated constants for calculation of the weight-average molecular weight of the captured HMWF. The collimated UV beam shall be placed so as not to be coincident with the light scattering optics. A photo-detector, possibly with a band-pass filter for the chosen wavelength, shall be placed across the cell from the UV light input and used to determine light absorbance at the chosen wavelength. Provision may be made for a reference beam path outside the cell. The UV absorbance signal will be utilized for determination of sample concentration in the light scattering cell. Alternative concentration sensors may be employed such as ultrasound, refractive index, IR etc. A sketch of the device is shown in Figure 2.





3. OTHER INFORMATION (e.g., test data, reduction to practice, planned use)

4. RECORDS:

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* Disclosure Letter should be addressed to Lab Manager of each inventor.

5. WITNESSES AND DATE

READ AND UNDERSTOOD BY:

_____
Witness

Date:_____

*INVENTOR_____
Signature

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Type Inventor's Name

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Laboratory or Program

Date:_____

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*When the invention is joint, all inventors should sign and date the disclosure letter.

(Complete and attach an Invention Disclosure Statement, Form RD-506A, for each inventor.)

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